THIRD QUARTERLY REPORT

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GODDARD SPACE FLIGHT CENTER

COVERING

ALKALINE BATTERY SEPARATOR STUDY

December 28, 1962 - March 28, 1963

Contract NAS-5-2860

The Carl F. Norberg Research Center The Electric Storage Battery Company Yardley, Pennsylvania

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TABLE OF CONTENTS

		O 40 3,	<u>Page</u>
I. "	Status Summary	• • • • • • • • • • • • • • • • • • • •	, 1
o	A. Introduction	• • • • • • • • • • •	
	B. Previous Results		1
II.	Work Accomplished During the Reporting Period	od	1
	A. Tensile Strength of Modified Cellulose		1
	1. Procedure		2
- ب	2. Results	, , , , , , , , , , , , , , , , , , ,	,
	Table 1	• • • • • • • • • • • •	
	B. Wet and Dry Strength Measurement of Me	mbranes	3
es"	1. Procedure		3
	2. Results		3
	Table 2		4
-	C. Oxidation Tests	<u> </u>	` , ` ;
	1. Sodium Hypochlorite Method	,	5
	2. Gaseous Oxygen Test	• • • • • • • • • • • • • • • • • • •	5
, , ,	a. Procedure	*	. 3
	b. Results		6

TABLE OF CONTENTS (Cont'd)

4	9 ° y	-	2 8	" ن	<i>,</i> .	<u> Page</u>
	Table 3,	•••••	; • • • • • • • • • • • • • • • • • • •			6
	3. Potassiur	n Perma	nganate (Test	· · · · · · · · · · · · · · · · · · ·	7-
12	a. Proc	edure 🔐	• • • • • • •	, • • • • • • • • • • • • • • • • • • •		
,						
, .		٠ , 6		* 0 · · · · · · · · · · · · · · · · · ·		4 .
II.	Investigation of C	commerc	ia! Sépar			8
,	A. Tests and Prod	j*)		_ + - +	• • • • • • • • • • • • • •	٠ ، ر
	B. Discussion of	Results	S	••••	• • • • • • • • • • • • • • • • • • • •	9
	Future Work			٠ , -	19	
	Literature Survey	بالحي ي	,			
<u>= /</u>	Appendix					
/II.	Errata		• • • • • • •			15
6	Tables 5 - 10	, , , , , , , , , , , , , , , , , , ,	,	, " () () () () () () () () () (

I. Status Summary

A. Introduction

This is the third quarterly report covering work during the reporting period December 28, 1962 through March 28, 1963 on Contract NAS-5-2860, which was awarded to The Electric Storage Battery Company by the National Aeronautics and Space Administration, Goddard Space Flight Center, on June 28, 1962.

B. Previous Results

In our previous report (ESB Report No. 7054.2) we compared the effects of aerobic and anaerobic hydrolysis in an alkaline environment on the degree of polymerization (DP) of cellulose. In an aerobic medium, the degree of polymerization of cellulose vs. time followed an irregular up and down pattern. In an anaerobic medium, the degree of polymerization followed a regular downward trend. It was concluded that oxidation caused the irregular trend and that it was the more important of the two reactions. In order to observe how far reaching the oxidative effect was, we decided to measure the tensile strength of cellulose hydrolysed in an anaerobic medium, and to compare the results with previous tensile measurements of cellulose hydrolysed in an aerobic medium.

II. Work Accomplished During the Reporting Period

This report presents tensile strength measurements of various membranes as well as experimental oxidation studies made during the reporting period.

A. Tensile Strength of Modified Cellulose

Tensile strength, or the breaking strength per unit cross section of a material, serves as an indication of the structural attributes of the material. During chemical treatment of a material, an alteration which would affect the structure of the molecule would in turn affect the tensile strength. Since cellulose is chemically altered during hydrolysis and oxidation, tensile measurements should indicate modifications taking place in the structure

of the molecule. We measured, therefore, the tensile strength of cellulose, which had undergone alkaline hydrolysis in an anaerobic medium, in order to ascertain the extent of chemical modification.

1. Procedure

Cellophane samples 3 in by 3 in were cut on an Accurate Steel Rule die. These samples were placed in one compartment of a double tube. Thirty milliliters of 40% KOH were poured into the other compartment of the tube. The tubes were frozen in liquid nitrogen, attached to a degassing apparatus, and sealed off under vacuum. The frozen KOH was melted and then it was poured into the compartment of the tube containing the cellophane. The tubes were placed in a constant temperature bath for designated time periods, and then withdrawn (This procedure is given in detail in the previous report ESB Report No. 7054.2). The samples were washed with water, 10% acetic acid, and water. The tensile strength was measured wet on a Thwing-Albert Tensile Tester.

2. Results

The following table shows a comparison of the tensile strength of cellophane subjected to aerobic and anaerobic hydrolysis at 90°C in 40% KOH.

TABLE 1

Hydrolysis of Cellophane in 40% KOH at 90°C

In Hour	. ר			e. Str	ength	in I		erobić
0	, ,	11.8	,	**	ر ا	.,	. 1	1, 8
24		10.8						7.7
48		10.1			.,	J		7.4
7.2	1)	3.6		,	J 1			5.8
96	-	6,5	U IJ		· ·			5, 5
Í 20		3.7			,		,	5.9
144	1	5.8			41		. 1	5,6
168		5.8	21	ų.	0	;	, ,,	4.5
192		≥ 4 . l ₀					,	5.2
216		8.3	F			· , .	,	3.7
240		.8.5			1			4.1
	0. 24 48 72 96 120 144 168 192 216	0 24 48 72 96 120 144 168 192 216	Aerobic 0 11.8 24 10.8 48 10.1 72 3.6 96 6.5 120 3.7 144 5.8 168 5.8 192 4.1 216 8.3	Aerobic 11.8 24 10.8 48 10.1 72 3.6 96 6.5 120 3.7 144 5.8 168 5.8 192 4.1 216 8.3	Aerobic 0 11.8 24 10.8 48 10.1 72 3.6 96 6.5 120 3.7 144 5.8 168 5.8 192 4.1 216 8.3	Aerobic 11.8 24 10.8 48 10.1 72 3.6 96 6.5 120 3.7 144 5.8 168 5.8 192 4.1 216 8.3	Aerobic 0 11.8 24 10.8 48 10.1 72 3.6 96 6.5 120 3.7 144 5.8 168 5.8 192 4.1 216 8.3	Aerobic Ana 0 11.8 1 24 10.8 48 10.1 72 3.6 96 6.5 120 3.7 144 5.8 168 5.8 192 4.1 216 8.3

The exclusion of air during hydrolysis has allowed hydrolysis to proceed in a normal manner evidenced by a regular lowering of tensile strength values. Hydrolysis in air results in tensile values which follow an irregular pattern due to the influence of oxygen on the reaction.

Figures 1 and 2 show the combined patterns of degree of polymerization (from viscosity measurements) and tensile strength in aerobic and anaerobic media.

B. Wet and Dry Strength Measurement of Membranes

The dry and wet tensil's strengths of some commercially available separator materials were measured.

1. Procedure

Samples of the materials were cut into 3 in, by 2 in, strips. Ten samples were measured dry to give an average tensile value. Ten other samples were soaked in distilled vater for fifteen minutes, and then tensile tests were made. The measurements were made on a Thwing-Albert Tensile Tester. Model 30 PM-24. The width of the jaws was 2 in, and the gage length 1 in. The machine speed was approximately 4 in, per minute.

2. Results

The following table shows the results of the tensile strength measurements on the various films:

TABLE 2

Tensile Strength in Pounds

*	Material		<u>Dry</u>		Wet
	9			Ċ	
ř.	Cellophane PUDO-300	•	40	•	12
~ 2	Cellophane PUDO-600	••	56		17
3	Avisco Gel Film	r	Z2	-	• 6
4	Fibrous Sausage Casing-Avisco	,	102		48
5 -	Silvered Sausage Casing-Avisco		110+ \$0	ti.	58
6	Permion, 300 - RAI		21		12
. 7	Permion 600 - RAI	· ·	.37	' ''	19
8	Polypor WA		97	•	79 -
9.:	PMA 95/5	,			3
10	MIPOR 12CN		8		8
11.	MIPOR 13CN	,	5 " .		5
12	MIPOR 34CN		9		9
13	MIPOR 34PN		16		16
14 0	Cellophane 111-1 DuPont		43		17
15	Cellophane 111-2 DuPont		48		16.
	•			Tr.	

For the cellophanes (1, 2, 3, 14, 15) the wet strength is approximately one-third that of the dry strength. For the other membranes, excluding the Mipor films, the wet strength appears to be one-half that of the dry strength.

The Avisco silvered sausage casing tested dry, reached the value of 110 lbs, the maximum load registerable on the machine. The wet tensile strength of the silvered film was 58 lbs. Since the wet strength values are the more important from a practical standpoint, a good comparison of the material strengths can still be made without deviating from a standard sample size. The Mipor membranes exhibited an identical dry and wet tensile strength. These membranes were not readily wettable. To increase their wettability, the Mipors were soaked in alcohol and then in distilled water. However, there still appeared no difference between dry and wet strength. Cellophane 111-1 and 111-2 were experimental films submitted by duPont as high-molecular weight cellophanes.

C. Oxidation Tests

Oxidation resistance was studied using three oxidants: Sodium hypochlorite, gaseous oxygen, and potassium permanganate. The potassium permanganate method was assessed to be the most useful method, because all membranes could be conveniently tested in it, and reproducible results could be obtained in a short time period.

1. Sodium Hypochlorite Method

Sodium hypochlorite was reported as an ideal oxidizing agent because it oxidized homogeneously, operating in an alkaline medium. However, the hypochlorite solution presented instability problems which we were never really able to control.

We tried to dilute the NaOCl solution using 40% KOH in dilutions of 2500 parts KOH solution/l part NaOCl solution. Even with this large amount of alkali present, the solution was not stable, as shown by analysis of the solution on a day-to-day basis. The reduction of the hypochlorite did not follow a regular rate and so it was impossible to establish a uniform rate for the spontaneous decomposition of the hypochlorite solution. We thus decided to discard the hypochlorite method.

2. Gaseous Oxygen Test

Our next attempt to develop an oxidation test was by bubbling oxygen through an alkaline solution containing the sample to be oxidized. This method is useful with the cellulosic materials, but requires excessive time for more inert separators.

a. Procedure

Samples of cellophane 8 in. by 4 in. 300 PUDO were placed in a gas washing bottle, tall form, with a fritted disc. They were then covered with 400 ml of 40% KOH. Oxygen gas flowed into the bottle at a constant rate of 14 cc/min regulated by a flowmeter attached to the tank. The tests were run at 25°C and 50°C for

various lengths of time. After removal from the bottle, the cellophane samples were washed with water, acetic acid, water, and dried. Solution viscosity tests were run to determine the degree of polymerization (DP) of the cellophane.

b. Results

The data obtained from the samples run at 25°C showed the same up and down pattern of DP measurements that was reported previously. At 50°C a regular curve was obtained over a three day period as shown in the following table.

TABLE: 3

Cellophane Hydrolysis with Gaseous Oxygen in 40% KOH at 50°C

Time in Hours		u.	Degree o	f Polymerizatio
0	* _ a	_		533° v
1	,	÷		5.26
- '16	,	-,		511
24			* 1	439
48	·		2	399
. 72	. 3	. · ·	,	372 ,,

According to the work done previously, the change in the degree of polymerization of cellophane in an alkaline environment in the presence of oxygen exhibited an irregular pattern. However, the results obtained by bubbling oxygen through KOH at 50°C follow a regular downward trend. In addition to the regular trend, the degree of polymerization values are also much lower than any reported for anaerobic conditions at 90°C after ten days, and for aerobic conditions at 60°C after three days. A comparison of the results obtained in the aerobic hydrolysis experiments, and those in which oxygen was bubbled through the solution, indicate that oxygen availability is the controlling factor in the viscosity curves offered.

3. Potassium Permanyanate Test

Since the hypochlorite method was found unsuitable, some other method was needed to assess the oxidation resistance. A method using potassium permanganate for testing alkaline battery separators was available, and was adopted to the current work. The extent of oxidation was neasured by weight loss.

a. Procedure

The material was dried for two hours at 80°C. A weighed sample of approximately one gram of the dried sample was placed in a 600 ml beaker containing 250 ml 5% KMnO₄ and 50 ml 25% NaOH. The weighed sample was forced under the surface of the solution by means of a glass rod. The beaker was placed, for one hour, in a constant temperature bath at 50°C. When the treatment was finish ed, the contents of the beaker were washed with distilled water until the disappearance of the red color. One hundred ml of a 5% oxalic acid solution, slightly acidified with H2SO4 (5 ml conc. H-SO4 to 1000 ml of 5% oxalic acid), were poured into the beaker, and then heated to 70°C. This process dissolved are manganese dioxide still adherent to the test piece. The sample vas washed with distilled water until the test for oxalate was negative. The sample was then dried to constant weight. The difference in weight between the original and the final weight was recorded as a percentage loss.

b. Results

Several membranes were subjected to permanganate oxidation and the results are recorded in the following table.

TABLE 4

KMnO₄ Oxidation for 1 hr at 50°C

Material	, .,	0 3	Per	cent Weid	ght Loss
MIPOR 13CN MIPOR 34PN	W (5)	e de la composición dela composición de la composición de la composición de la composición dela composición de la composición dela composición dela composición de la composición dela composición de la composición dela c	, · · · · · · · · · · · · · · · · · · ·	0.8	S
Polypor WA PMA 95/5 Silvered Sausag	ae Casing			7 16 31	
Fibrous Sausag			o"	38	1 ,

Cellophane 600 PUDO was subjected to the KMnO₄ treatment at room temperature for 1 hour. The film crumbled upon removal from the beaker. The weight loss was measured as greater than forty parcent.

This test has given us reproducible results in a relatively short period of time. It can be used, with modifications, on all films. Other films will be subjected to this test in order to compare their relative oxidative resistance.

III. Investigation of Commercial Separators

In addition to the twenty-one separators listed in the preceding quarterly report; the following separatorematerials were studied:

- 22% Cellophane 111-1
- 23. Cellophane 111-2

These are experimental cellophanes, made by E. I. duPont de Nemours and Co. They are presumably regenerated cellulose of high molecular weight. Neither material is in commercial production.

- 24. Fibrous Sausage Casing (Visking)
- 25. Impregnated Cellophane

This is dipont's cellophane PUDO-300 (identical with sample No. 1), impregnated with m-phenylenediamine, as described in our 2nd Quarterly Report No. 7054, 2 (page 8).

26. Acropor WA - a weak acid, acrylic ion exchange resin, made by Gelman Instrument Co. Preliminary tests had indicated that it was sufficiently different from the Polypor WA (made by Niemand Bros. before transfer of operations to Gelman) to warrant a separate study.

A. Tests and Procedures

These materials were subjected to the same tests as the original samples, i.e.

- 1. Electrolyte absorption and retention (at 26°C).
- 2. Electrical resistance after different spaking times.
- 3. Electrical resistivity, after 24 hours soaking.

The procedures used for the individual tests have been described in the 2nd Quarterly Report.

In addition, the dimensional stability of all samples after various soaking times, at three temperature levels (room temperature, -28°C and +90°C) was investigated. The procedure was as follows:

Samples 3 x 1 inches were used in the early work; later, the sample size was increased for the sake of greater sensitivity in measuring changes. The samples were die cut by means of a steel rule die (Accurate Steel Rule Die Manufacturers, New York). Changes in length and width were measured with a stainless steel ruler. The thickness, before and after the tests, was measured with a stainless steel indicating gauge (Ames, Model 262), having a foot 0.500" in diameter and loaded at 5 oz/sq. in. The thickness of each sample was measured in several places and the results averaged.

For testing at 90°C, the samples were covered with 31% KOH in cylindrical stainless steel pots. 2.5 in. high and 3 in. diameter, equipped with tight-fitting stainless steel lids (to minimize evaporation and $\rm CO_2$ absorption). The closed pots were placed in an oven, controlled at 90 \pm 1°C.

The tests at the lower temperatures were carried out in plastic containers (modified acrylic) which were also equipped with lids. The samples which were to be tested at -28°C were placed in a refrigerator, maintained at this temperature.

B. Discussion of Results

Tests results are summarized in Tables 5 through 10.

The <u>dimensional change</u> measurements were carried out for four weeks. All but one of the materials tested (a cellophane) survived the soaking at -28°C for four weeks. Several cellophane samples were too much deteriorated after four weeks in 31% KOH at room temperature to permit a reliable measurement of their thickness. The remaining samples, including some cellophanes, could still be measured.

None of the materials survived more than three weeks in KOH at 90°C.

The new experimental cellophanes show slightly higher electrical resistivities than commercial grades but the differences are very minor. Both exhibit slightly less swelling in thickness but both absorption and retention of the electrolyte are markedly lower than for commercial grades.

The amine treatment also caused a substantial decrease in electrolyte absorption and retention of a commercial cellophane (cf. Nos. 1 and 25), but it resulted in a moderate lowering of the electrical resistivity which was low to begin with (from 8.45 to 7.8 ohm-cm).

Visking Fibrous Sausage Casing shows an absorption similar to the grades tested before. While the retention appears somewhat lower, its resistivity is of the same order.

Absorption and retention values for Acropor WA were approximately 40% lower than for its predecessor, Polypor WA. At the same time, however, its electrical resistivity was also substantially lower (around 12 ohm-cm for Acropor, against 20 for Polypor). This brings its resistivity close to that of cellophane.

IV. Future Work

The remaining tests on the dimensional stability are under way and will be ingluded in the next quarterly report.

Studies of the oxidation resistance of currently available separators are continuing and are to be extended to impregnated and modified materials.

Modifications of existing materials by suitable impregnants, by crosslinking of cellulose and by graft-polymerization will be studied.

All equipment and materials for an investigation of silver ion migration by means of radioactive tracer techniques have been received. These studies should be completed during the next reporting period?

V. Literature Survey

The survey of the available literature was continued. New patents and the published literature were covered, as well as reports to the U.S. Governmental agencies on work done under contract by various organizations.

Extensive work on the development of new separators was done at the Polytechnic Institute of Brooklyn during 1951-1957, under the direction of Drs. P. F. Bruins and H. B. Gregor, under contract with the U.S. Army Signal Corps (Contracts No. DA-36-039SC5425 and DA-36-039SC42578). A large part of this effort was directed towards the development of separators for silverzinc batteries. Three main lines of approach were followed:

- 1. Modification of cellulose.
- 2. Polymeric film2type membranes.
- 3. Ion exchange resins, dispersed in film-forming polymers.

One of the numerous experimental membranes developed in the course of this work is in commercial production. It is made by dispersing a hydrophylic polymer, e.g. poly (vinyl alcohol) in a film-forming matrix sucg as Dynel (a vinyl chloride-acrylonitrile copolymer). It forms the subject of U.S. Patent 2,884,387 (see 2nd Quarterly Report, pp. 12 and 25) and is presently manufactured by Gelman Instrument Co. under the tradename "Acropor" (previously "Polypor", made by Niemand Bros.)

All the work at the Polytechnic Institute of Brooklyn, carried out between 1951 and 1957, is embodied in 26 Quarterly Reports to the Army Signal Corps. Parts were also reported in the chemical literature: an article by Shair, Bruins and Gregor(1) described the use of maphenylenediamine, impregnated on a microporous membrane and crosslinked by formaldehyde. Another paper(2) deals with separators made by casting polymeric films with finely distributed ion exchange resins. Individual phases of the project have been the subject of a number of theses by students of the Institute.

Other new separators, or novel forms of older separator materials, were described in the patent literature. A search of the patent literature produced only two new U.S. patents. In addition, a number of Japanese patents describe separators for the silver-zinc system. Some of these tread familiar ground but others employ novel compositions, e.g. mixtures of poly (vinyl alcohol) with poly (furfuryl alcohol), poly (vinylpyridine) or poly (vinyl pyrrolidone), or copolymers of vinyl alcohol with vinyl pyridine or vinyl pyrrolidone. These additional patents are listed in the Appendix.

Evaluations of various combinations of previously known separator materials are described in several reports to Governmental agencies. These are principally the following:

Delco-Remy Division of General Motors Corp.

Final Report ASD-TDR-62-668 under Air Force Contract No. AF33(600)-41600 (October 1962).

Missile Battery Division, Electric Storage Battery Company

Final Report E-6-63 under NASA Contract No. NAS-5-1607 (March 21, 1963).

Earlier reports by the Eagle-Picher Co. and by Gulton Industries were also consulted.

Lite ature References

- 1. Shair, R. C., Bruins, P. F., Gregor, H. P., Ind. Eng. Chem. 48, 381-5 (1956).
- 2. Bieber, H. H., Bruins, P. F., Gregor, H. P., Ind. Eng. Chem. 50, 1273-8 (1958).

VI. APPENDIX

Patents Pertaining to Separators for Silver-Zinc Batteries

U. S. Patents

- 3,049,578 Appl. 3/12/56, issued 8/14/62 E. Jonsson, S. U. Falk (to A/B Jungner) Cellophane bags.
- 3,071,372 Appl. 2/17/60, issued 3/12/63 D. G. Soltis (to Union Carbide Co.) Heat sealable separator made by coating paper with a mixture of poly (ethylene oxide) and methylocellulose.

Japanese Patents

- 1470/59 Issued 3/18/59 T. Fukuda. S. Oda (to Matsushita Electr. Ind.) Cellophane separator, treated in a bath containing formic and hydrochloric acids (to improve alkali resistance).
- 1824/59 Issued 4/6/59 M. Fukuda, T. Iwaki (to Matsushita Electr.

 Ind.) Poly (vinyl alcohol) separator, treated in a bath containing formic and sulfuric acids (to improve chemical resistance) and sodium sulfate (to reduce electric resistance).
 - 2764/59 Issued 4/21/59 S. Matsuno (to Yuasa Battery Co.) Separators with improved resistance to alkali
 - a. Parchment paper, coated with a rosin.
 - b, Filter paper, impregnated with poly (vinyl alcohol).
 - c. Microporous rubber, impregnated with ethyl cellulose.
- 9069/60 Issued 7/13/60 K. Osawa (to Jap. Storage Battery Co.) "Silver cellophane", made by immersion in AgNO₃ solution, then formaldehyde treatment to reduce AgNO₃ to colloidal Ag.

- 18658/60 Issued 12/26/60 M. Fukuda et al (to Matsushita Electr. Ind.)
 Copolymers of vinyl alcohol and unsaturated alcohol, in sheet form, used as separators.
- 18659/60 Issued 12/26/60 M. Fukuda et al (to Matsushita Electr. Ind.)

 Mixture of poly (vinyl alcohol) and furfuryl alcohol, cast into sheets (supported or unsupported) to form separators.
- 18660/60 Issued 12/26/60 M. Fukuda et al (to Matsushita Electr. Ind.) Mixture of poly (vinyl alcohol) with poly (vinyl pyridine) or poly (vinyl pyrrolidone).
- 18661/60 Issued 12/26/60 M. Fukuda (to Matsushita Electr. Ind.)

 Vinyl alcohol is copolymerized with vinyl pyridine or vinyl pyrrolidone.

VII. Errata

Reight 7054.1 (First Quarterly Report) ? p. 6, last paragraph, 1. 4:

For: "...in going from the hydroxyl group (17g) to the carboxyl group (33g)..."

Read: "...in going from the CHOH group (30g) to the carboxyl group (45g)..."

Report 7054.3 (Second Quarterly Report) p. 1,5, last paragraph, 1.1:

For: "active" read "above"

p. 18, last paragraph, 1.1:

For: "mechanical" read "dimensional"

p. 25, 1.3%

For: "4/28/50" read "4/28/59"

TABLE 5

Electrical Resistance (Ohms) in 31% KOH at 26°C After Different Soaking Times

(Average Values for 3 Samples Each)

Sample N	o.ºº Materia	1	A	5 min."	10 min.	20 min.	1 hr	24 hrs.
., 22	Celloph	ane lll-l	(duPont)	0.087	0.084	0.083	0.083	0.076
23		ane 111-2		0.093	0.086	0.085	0.082	0.079
24	Fibr. Sa	usage Cas	ing (Visking)	0.166	0.156	0:"158"	0.154	0.172
25-	" Celloph	ane PUDO-	300, impregn	. 0.084	0.069	0.071	0.068	0.065
26	Acropor	WA	7	0.280	0.268	0.224	0.190	0.156

TABLE 6

Electrical Resistance in 31% KOH at 26°C

Sample No.	"Material	R_0	% Devia- tion	Dry Th	ickness 4 cmx10	Wet Th inxl0	icknes 4 cmx10	54 Chang	R _i	ρ
22	Cellophane 111-1	. 076	3.9	12	30	29	74	+139	.011	. 9.9
23	Cellophane 111-2	.079	8.6	12"	30	30	76	+150	.012	10.0
, 24	Fibr. Saus. Casing	a. 172	5, 2,	41	104	80	203 ا	o + 95	.026	8.2
25	Cellophane PUDO-300	.065	3.0	10	25	32	81	*+ 220	.010	7.8
26	Acropor WA	0.156	23,3	41	"" j104	50	1 27	+22	. 0 23	11.0
, , ,	Column No.	· 1	2	·3	4	5 ,	6.	" ' 7 "	. 8	9

<u>Legend:</u>

Column 1: R_0 = Observed values (ohms), resistance after 24 hrs. soaking in 31% KOH, exposed area 0.15 sq. in. (average of 3 samples).

Column 2: Average % deviation of the 3 samples of Column 1.

Columns 5

and 6: Thickness of separator after 70 hours soaking in 31% KOH.

Column 7: % Change shows swelling (+) or shrinkage (-) of separator thickness after 70 hours soaking.

Column 8: R₁ = Resistance (ohms for 1 sq. in.) at actual thickness.

Column 9: $\rho = Specific Resistivity (ohms - cm)$.

TABLE 7

Electrolyte Absorption and Retention at 26°C

		. 17					1.					
-	Sample	Dry Weight"	Thick	nesș	. A	ol.	Absorpti	on	442	Rete		า 🤺
	No.	grams	cm_x	10-4	cm³ :	x 10 - 4	° ′g	g/cc		g "	,	g/cc
.)	-		Dry	Wet	$\overset{\bullet}{\mathop{\mathbf{D}}}$ ry	Wet			•			
							O.		,	,		5 . 9
٥.	ິ a° 2:2	0.1580	28	74	7108-	-887	0.5560	5.2	ω'	0.2151		2.0
0	23 ′	0.1531	38	76	147	- 294	0.6965	.,4.7		-0.2854		1.9
	.24	0.2894	81	· 203	314	887	1.0144	3, 2,		0. 5578	12	1.8
-,	25	0.0859	19	48	93	194	0.3521	3.8		0.2103		2,3
	,26 -	0.1061	48	123	23.6	~252	0.3210	1.3		0.1556	_ '	0.6

TABLE 8

Dimensional Changes in 31% KOH at -28°C

			(ı		•		,
			70 hours	- , ,	7	days	
\- A ₀		Length	Width Th	ickness I	erigth	Width T	hickness
1	Celiophane PUDO-300	- 5	+ 10	+3 00	-5	, 1 10	+ 300
2 ·	Cellophane PUDO-600	0	+15	†440°	0	+ 15	+ 360
3	Cellophane 140-PUT 76	5	+ 15	¥ 290 /	5	1 15	+ 280
4	Cellophane (Avisco	· 5	" + 10	+ 300	- 5	1 10	+ 290
5	Cellophane (Avisco)	+ 15	+ 60	+ 365	† 15	1 55	+ 370
6	Cellophane (Avisco)	0	+ 5	+ 240	0 °	0	+ 240(الم
7	Sausage Casing (Avisco)	÷ 10	+60 -	+ 350 "	+ 10	465 ·	+ 355
`8	Fibra Saus, Casing (Avisco)	- 10',	0	+ 650	0	0	+ 650
9	Silvered Saus. Casing	0	÷ 40	+ 460	0 -	+45	+410
10	Permion 300	0	, + 10	+ 180	+ 5	+10	+ 170
11	Permion 600	~ 0	+ 5	1 35	0	+ 5	+ 10
12	AMFion C-60	0	0 ⁷	+10,	0	0,	+ 10 *
13,	AMFion C-103	0	0	0 0	0	0	0
14	Polypor WA	0, ,	0	 5	0	0	+.5 . °
15 - "	PMA/Polyethylene	-Tests no	t complet	e.			-
16	PMA/Polyethylene	Tests no	t complet	e.			11
17	PMA/Polyethylene	Tests no	t complet	e.		11	
18	Mipor 12CN	0	0 · · ·	0	,0	0	0
19	Mipor 13CN	0	0 .	∔ 5″	0	0	+5
20	Mipor 34CN	0	, 0	O .	0	0	.0 o
21	Mipor 34PN	0 .	0	0.3	0 .	0	0
22	Cellophane 111-1	. 0	+ 20	+240	0	+ 20· "	+.200
23 ,	Cellophane 111-2	0	+ 20	. + 2 50	0	+ 20	·+ 230
24	Fibr. Saus. Casing (Visking)	-90	0.	+300 · "	-115	0.	~+ 260 ° ·
25° c′	Cellophane PUDO-300, impregn	ated with n	n-phenyle	nediamine	- Test	s not co	omplete.
26-	Acropor W -	*	•	· ·		ts not c	
-	- "					,	- Table

Legend: 0 denotes less than 5% change

* Test discontinued due to sample deterioration.

TABLE 8 (Cont'd)

Dimensional Changes in 31% KOH at -28°C

		. 1	ii .	Sec. 82 (25)	i			· · · · · · · · · · · · · · · · · · ·	
. '	5	14 day	<u>s</u>		21 days	3		28 days	(•
11, "	Length	Width	Thickness	Length	Width	Thickness	Length	www.ch.Ti	hicknes
	: ,	· 1°		$\mathcal{F}_{ij} = \mathcal{G}_{ij}$	' []	-		1 2	
1"	-10	→ +10/	+330	-10	10	+ 280	-10	+10	+350
. Z	., 0	+15.	+380	. 0	†15,	÷ 420	*	+15	+,420
3	- 5	+ 15	+ 27 5	-5	+10	e + 3 _, 30	∤−5	+10	÷300
4	− 5	+10	+320	- 5	1500	o + 400	- 5	,+10° °	+360°
ž	+ 10	÷50°	+340	*15°	†55	+345	+ 10	+ 30 .	+320
6	.,0	0:	.³+275	0	þ "	+ 265	> 0	+5	±300
7	÷ 10	+65	+400	+15	- 70	+ 330	- 5 幸 - 4	∜c	+345.
8	- 10	🥕 👸 🔞	+675	∞ ~-10	þ	+700	-10	, 0 , ,	+800
920 0	² 0 + ¹	+45	+430	0	45	+ 385	0	+55	+395
10 %	, + 5	+10	+160	+ 5	10	*	+ 5	+10	水
11 W	0	Ĵ + 5	+10	0 % 0	0	, 0	0	≯15	0
12	0	0	+10	0	0	+10.	<i>~</i> ∤0	ູ0 -	+15
13	0	0	, 0 ≥ .	0	0	0	0	0	0~
14	0 ,	0	+5	0	- 0	+5	0	○ 0 ,	+25
. 15 🔭 🦠	Tests	not comp	lete.	w "		<u>.</u>	· (- , -		2
16 .	-	not comp	4	•	J ., 3		1		
17 =		not comp	•		. ~	*	, T	,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	15
18	. 0	0	0 ,	, , , , , , , , , , , , , , , , , , ,	. 0	+5	O // =	. 0 ر	+5
19	0	0	+5,	· 0	. 0	" _~ +5	$\langle 0 \rangle$. 0 →	+10 "
20	0	.0	0 ~	. 0	0	+10	0/.	.,0	+15
21	0 ີ	0	0 "	0	-0	"。 0	٠ <u>٠</u> ا	. 0	- 5
22	+353	+,25	+200	+ 5	+25 "	+250	*+ / 5	+20.	+250
23	0	+20	+220	. • • 0 , . * *	+20 "	¥300	. #5	.+20	+370
24	~115 "	÷ 0	¥260 ×	-10	0	+280	/ - 10	0.	+330
25	Test's	not comp	' 17	~ u, g	3	-	- 1	es.	
₹ 26		not comp	,		· ·			٠, '	,

TABLE 9
% Dimensional Changes in 31% KOH at 26°C

	* * * * * * * * * * * * * * * * * * * *	, ,		· ·		,	**,.
į		. 0	70 hour	<u>s</u>	, ,,	7 days	`
1	the transfer of the state of th	Length	Width	Thickness	Length	Width	Thickness
				±			-
1 "	Cellophane PUDO-300	25	+5`'	+280	-5	ે +5	+200
2 ;	Cellophane PUDO-600	0	+5	+160	. 0 :	+ 5	165
.3 ,-	Cellophane 140-PUT 76	0	÷ 5	+170	0 ~	+5	J+170
4 ,. 4	Cellophane (Avisco)	-5	+5	+190	250	+5	+280
·5 ·	Cellophane (Avisco)	0. €	+10	+160 -	0	+10	+160 ~
6	Cellophane (Avisco)	0	+5	+175	Ö	0	+190
> 7 *	Sausage Casing (Avisco)	0 .	÷10 = 41	+175	0	÷10	+170
8 .	Fibr. Saus. Casing (Avisco)	ગ ં~5	+5	+170	- 5	0	+180.
9	Silvered Saus, Casing		· +5	+ 6 0 .	5)	+5	+160
. 10	Permion, 300 - 19 19 19 19 19 19 19 19 19 19 19 19 19	. + 5	+10	+25	+5	+10,	ε 0 🤔
. 11-	Permion 600	. 0	0 .	+ 25	0 -	, 0 .	÷25
l'a	AMFion C-60	0 ' "	0 -	+15%	0.	0	+10 ~
13	AMFion C-103	÷ 0	0 -	- 5	- 0	. 0 -	= -10 : >
14	Polypor WA	0	. 0	; - 5	.0	- 0	0
15	PMA/Polyethylene	Tests	not compl	ete. 🔧		0	•
1.6	"PMA/Polyethylene"	Tests	not compl	ete.		· , ,	9
17	PMA/Polyethylene	Tests	not compl	ete.	= "	,	
18	Mipor 12CN	0	0	0	0	0	0 ,
19 .	Mipor 13CN	0	` O	, 0 ,	Q.	0	0
200 ≥ 4	Mipor 34CN	%;3 0 -	·0 0	0	. 0	0 (_` +5
21	Mipor 34PN	0 -> 0	. 0	0 -	0	0	0
22 .	Cellophane 111-1	0 .	° 410 °	+150	` 。 0	+10	+140
23	Cellophane 111-2	0	# 5	+150	Ų	+.5	+150
24 。	Fibr. Saus. Casing (Visking)	_,⊹ ~5 °°°	0	£120	−5 °	O . ~	+95
25 _,	Cellophane PUDO-300, impreg	Tests	not compl	ete.		.	2
26 "	Acropor WA		not compl			·	
9			, v - ~ ~,		,		~ · ·

Legend: 0 denotes less than 5% change.

^{*} Tests discontinued due tousample deterioration.

TABLE 9 (Cont'd)

% Dimensional Changes in 31% KOH at 26°C

		14 day	14 days		21 days			28 days		
	Length	Width	Thickness	Length		Thickness	Length		Thickness	
1.	· · · · · · · · · · · · · · · · · · ·			ė.	<u>.</u>	4.		٠,	٠	
1	÷ 5	+5	+170	**	*	* *	*	**	*	
Z	0	+5	+180	°0	*	÷180	* .	5 ×	*	
" 3	- 5	+5 ,	+175	* 🧳	*	*	\	* 0	*	
4	* 5	+10	+280	2,4	*	华	*	* -	*	
5 ″	0	+10	+160	0	+ 10	+ 160	0	+10	+160	
6	5	*	ر ع اد "	*	*	*	**	*	*	
7	0 .	+100	" +160	0	+10	+160	0	÷10	-175	
8	, Ö:	[⊕] 0	+19C	0 .	0	+190	-10	0	+200	
9	-5	Ö	+180	-5	÷5	+180 (**		+5	+200	
° 10	+ 5 2	+10	+10	+ 5	+10	+10	÷ 5	+10	+50	
11	0	0 .	+20	0	0	+20 ~	Ó	0	+20	
12	0 1	0	. +10 .	0 1	0	+10	¹ 0	0	÷15	
13	·	0.	0 ~	0	0 '	0	· *	*	+5	
14	0 -	0	+5	0.	0 .	+ 10	0.	0	+30	
15	l'ests no	t compl	ete.	1,0		, ,	•	~		
16	Tests no	ot compl	ete.	×	<i>-</i>	-		•	•	
17	Tests no	t compl	ete.				-		,	
18	0	, Ò	0	. 0	0 3, 3	. 0	0	0	″ . +5	
19	0	0 .	+5	. 0	0	+5 `	0	0	+15	
20	0	0	+5	0	. 0	+5	0 .	0	+15 - ["]	
21	. 0	0 .	. 0	0	0	0	" 0	. 0	*5.	
22	0 , .	±10 ′	+140	- 0	+10	+140	*-	*	*	
23	. 0	₊ 5	"+150"	, O _{G **} "	*	+150	0	*	+150	
24	-10	0	+95	-10	0 ,	+95	-10	0 -	,, + 1,10	
25	Tests no	ot compl	ete.		_ V	• • .			•	
26	Tests no	_		*,	-			1 1		

TABLE 10
% Dimensional Changes in 31% KOH at 90°C

	-		70 hou		-	7 days	
		Length	Width	Thickness	Length	Width	Thickness
1	Cellophane PUDC-300	- 25	+ 15	+280	-25	+10	+300
2	Cellophane PUDO-600	- 20	0	+2,60	-25	.0	, +250
3	Cellophane 140-PUT 76	-15	0	+220	-15	0	+200
4	Cellophane (Avisco)	- 20	+ 5	*+336	-20	["] 0	+300
5 4	Cellophane (Avisco)	-15	0	+210	-15	+5	+195
6	Cellophane (Avisce)	-10	-5	÷240	*	*	* *
7	Sausage Casing (Avisco)	- 20	:+ 10	+250°	5 -20 '	÷5	+235
้อ	Fibr. Saus. Casing (Avisco)	- 1.0	0	+220	-15	-5	+195
9		Q -10	0	+230	-15	-10	+210
10	Permion 300	.0	0	-10	0 .	0	- 20
11	Permion 600	- 5	+ 10	+45	10	÷ 5	+50
. 12	AMFion C-60	0. **-	0	+5	. 0	0	+5
13	AMFion C-103	- 0	. 0	0	0	0	0
14	Polypor WA	. 0	₂ 0	+15	0	0	. 0
15	PMA/Polyethylene	Tests	not con	nplete.		•	¥
16	PMA/Polyethylene	Test's,	not con	nplete.	•		i le
17	PMA/Polyethylene	Tests	not con	npleté.			
18	Mipor 12CN	0	0	+15	-5	0	. +5 · .
19	Mipor 13CN	, O	0	415	-5	- 0	+10
20	Mipor 34CN	-15	. +10	+35	- 1.5	+5	+40
, 21 🤞 .	Mipor 34PN	- 15	+5	-15	-15	+5. °-	+30
•	Cellophane 111-1 (DuPont)	- 5	+5	+260	-15	* - ',	-230
23	Cellophane 111-2 (DuPont)	-10	+5	+120	-15	/ * .	+120
24	Fibr. Saus. Casing (Visking)	15	-10	+210	-15	-15	+,210
25	Celiophane PUDO-300, impreg.	•	not con	nplete.	•	5 `	
26	Acropor WA		not con	-		.,	·
		,	· ·	-, ·	•		والمستعلقة المستعلقة

Legend: 0 denotes loss than 5% change

^{*} Tests discontinued due to sample deterioration.

TABLE 10 (Cont'd)

% Dimensional Changes in 31% KOH at 90°C

2 1	,	14 day	ノ		21 day	S
ž **	Length		Thickness	Length	Width	Thickness
ч	2	2 %	~''s -			,
1	-25	+ 15	+310	-30	°+ 5,	+260
2 .	- 20	- 5 "	+260	-30 -	~ °5 🤌	+280
3 °	- 20	- 5	+200	-25	-10	+170
4	- 25	0	+3 20	30	-5	, +300 °
5	- 20	0	+ 27 5	*	*	3/4
6	· · · · · · · · · · · · · · · · · · ·	*	*	*	3,c	* a
7	- 25	*	+300	* * *	- ,*	* '
8	-15	-10	+240	-15	-10	+170
9 .	- 20 .	- 5	+270	-1,5	+5	+170
10	0 ຶ	+ 5	+20	0 ′′.	+5	0
11	-10 °	0	+36	-10	- 5	+ 22
12	0 -	0 .	" · 0	0	0 /	, 0
13	± 5 , ",	+ 5	· · • 0	**	<i>)</i> **	* -
14	0	0 .	2k	´ *¦¢	/ *	*
15	Tests	not com	plete.	/	٠	
16	Tests	not com	plete.	1.	. ś .	
17	Tests	not com	plete.	4 2, 4		
18	-10	0 ,	. +5	÷ ~-15	0	+25
19	- 20	Q 2.	+ 20	, / - 25 °	. 0	+10
. 20 "		4+ 5	+45	* ,	" **- ,	*8
21	~ 20	*	+90	*	* ,` _	, *
22 .	*	×.	+220	* .	*	*
23 $_{\odot}$	1 -15	* 5	+110-	- × ·	* * ,	*
24	. · _ 20	-15	+200 -	~ * ~ ,	3/4	* 3
25	Tests	not com	plete.	V		·
26	Tests	not com	plete.	, v	\ <u>.</u>	



